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Environmental Effects on Friction and Wear of Diamond and Diamondlike Carbon Coatings

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ABSTRACT

Reciprocating sliding friction experiments were conducted with a natural diamond flat, diamond films, and low- and high-density diamondlike carbon (DLC) films in contact with pin specimens of natural diamond and silicon nitride (Si_3N_4) both in humid air and in dry nitrogen. The results indicated that for natural diamond pin contacts the diamond films and the natural diamond flat were not susceptible to moisture but that moisture could increase both the coefficients of friction and wear factors of the DLC films. The coefficients of friction and wear factors of the diamond films were generally similar to those of the natural diamond flat both in humid air and in dry nitrogen. In dry nitrogen the coefficients of friction of the high-density DLC films in contact with pin specimens of both diamond and Si_3N_4 were generally low (about 0.02) and similar to those of the natural diamond flat and the diamond films. The wear factors of the materials in contact with both natural diamond and Si_3N_4 were generally in the ascending order of natural diamond flat, diamond film, high-density DLC film, and low-density DLC film.

Moisture in the environment increased the coefficients of friction for Si_3N_4 pins in contact with all the materials (natural diamond flat, diamond films, and DLC films). This increase in friction is due to the silicon oxides film produced on the surface of Si_3N_4 pins in humid air.

1. INTRODUCTION

As is well known, because diamond is hard and nonreactive, it is one of the slipperiest materials, and is similar to polytetrafluoroethlylene in terrestrial environments.[1] Natural diamond or high-pressure and high-temperature manufactured diamonds are available as three-dimensional products. However, the new materials, chemically vapor-deposited diamond films and diamondlike carbon (DLC) films, are available only in planar film or sheet form. With the diamond films and the DLC films a whole new range of products will be able to take advantage of the properties of diamond. Diamond films and DLC films offer tribologists and designers the combined properties of hard coatings and solid lubrication. For the fabricator or end user this new technology means an extraordinary expansion in tribological applications.[2]

Before diamond films or DLC films can be evaluated in practical bearing applications, it is necessary to demonstrate that they indeed have friction and wear properties and environmental stability similar to those of natural diamond in a variety of environments, such as humid air, dry nitrogen, and vacuum.[3] Recently several tribological studies of diamond films and DLC films have been reported).[3-11] Some work concentrated on the frictional behavior at high temperatures and in a vacuum, spacelike environment.[8,9,12] In the case of DLC films, plasma deposition processes have been used successfully to obtain very low-friction coatings that are stable to 500 °C in vacuum.[12]

Solid lubricating films designed for spacecraft applications should have the following three properties: low (0.1) coefficient of friction, good durability, and stability in both vacuum and terrestrial environments.[13] The environment to which a solid lubricating film is exposed can markedly affect its tribological properties, such as friction and wear life.[13,14]

The objective of this study was to investigate the friction and wear behavior of diamond films and DLC films in contact with a natural, single-crystal diamond pin and with silicon nitride (Si_3N_4) pins in humid air and in dry nitrogen. This study in particular addressed the effect of moisture on the friction and wear behavior. Comparative experiments for friction were conducted with a natural, single-crystal (111) diamond flat in the same manner.

2. MATERIALS

2.1. Diamond Films

Fine-grain diamond films were formed on flat surfaces of silicon carbide and silicon substrates by a microwave chemical vapor deposition technique that used a mixture of methane, hydrogen, and oxygen. A substrate temperature of 860 °C was used. Diamond deposition appeared to be thicker on the center of the silicon wafer (10.16 cm in diameter) than on the edge. The calculated thicknesses based upon light interference fringe measurement and Rutherford backscattering (RBS) were 0.5 to 1.5 μm, respectively, on the edge and center of the wafer. The deposition rate was estimated to be 0.14 μm/hr over the center area of 2.5 cm in diameter. The deposition and physical characterization of fine-grain diamond films are described in detail in Ref. 15.

Briefly, the fine-grain diamond films contained about 2.5 at.% hydrogen (by proton recoil analysis) and 97.5 percent carbon (by the RBS technique). There were no impurities other than hydrogen. The x-ray photoelectron spectroscopy (XPS) analysis of the diamond films indicated that the film surfaces contained predominantly C-C and CH_n bonds with small amounts of C-O and C=O bonds. Furthermore, the Raman spectrum of the film clearly showed the presence of a peak characteristic of the diamond at 1333 cm^{-1}. The size and orientation of the diamond grains were determined from bright- and dark-field electron microscopy and x-ray diffraction, and the grains in the film were found to vary from 20 to 100 nm in size, with the preferred orientation growth of ⟨110⟩. Optical transmittance of this free-standing diamond film was about 58 percent over the infrared region of 2.5 to 10 μm. The average surface roughness of the diamond films, as measured by a surface profilometer, was 15-nm root-mean-square roughness and 13-nm centerline-average roughness.

2.2. Diamondlike Carbon Films

DLC films (250 to 280 nm thick) were formed on flat surfaces of silicon nitride and silicon substrates by using the 30-kHz alternating-current glow discharge of a planar plasma reactor. The growth and physical characterization of DLC films are described in detail in Refs. 16, 17, and 9.

Briefly, the hydrogen concentration in the carbon films decreased in the range 7.7×10^{22} to 7.2×10^{22} cm^{-3} when the deposition power was increased from 25 to 300 W. The hydrogen concentration gives an approximate value of 0.8 for x in the formula CH_x. Furthermore, the average Vickers microhardness for the DLC films deposited on the Si_3N_4 substrates varied monotonically from 22 to 29 GPa as deposition power was increased. The higher the plasma deposition power, the greater the film density and the hardness.

2.3. Natural Diamond and Si_3N_4

Natural diamond was used in the reference experiments. The (111) plane of the diamond was parallel to the sliding interface. The diamond specimen was in the form of a flat platelet and had a mean surface area of approximately 30 mm^2. The flat surface of the natural diamond was polished with 1-μm-diameter diamond powder and with 1-μm-diameter alumina (Al_2O_3) powder and then ultrasonically rinsed with ethyl alcohol before each friction and wear experiment.

Natural diamond and hot-pressed, polycrystalline, magnesia-doped Si_3N_4 were used as the pin materials in the sliding friction experiments.[10] The hemispherical tip (1.3 mm in radius) of the natural diamond pin specimen and the hemispherical tips (1.6 mm in radius) of the silicon nitride pin specimens were polished with 1-μm-diameter diamond powder and with 1-μm-diameter Al_2O_3 powder and then ultrasonically rinsed with ethyl alcohol before each friction and wear experiment.

3. EXPERIMENTAL PROCEDURE

Reciprocating sliding friction experiments were conducted with the flats (the natural diamond, diamond films, and DLC films) in contact with the pins (natural diamond and silicon nitride) in humid air at a relative humidity of approximately 40 percent and in dry nitrogen. Details of the friction and wear apparatus and the experimental procedure are described elsewhere.[10] In each experiment a newly polished surface of pin specimen (natural diamond or Si_3N_4) and a newly polished flat surface (natural diamond) or a newly coated surface (diamond films and DLC films) of a flat specimen were used. In the desired environment the pin and the flat surfaces were brought into contact and loaded.

In order to obtain consistent experimental conditions, contact was maintained for 15 min before sliding. The reciprocating sliding friction experiment was then begun at a load of 1 N at room temperature (Table I). The pin traveled back and forth, retracing its tracks on the flat. Sliding velocity was 86 mm min^{-1} over a track length of 3 mm. The friction force was continuously monitored during a friction experiment.

Wear groove dimensions were used to calculate the wear volume of coating removed, as well as the wear factor for the coating. The wear volumes of coating were obtained from stylus profilometer tracings across the wear tracks in at least five locations, and then the average cross-sectional area of the wear track was multiplied by the wear track length (approximately 3 mm). The wear factor is defined as the volume of material removed in unit applied load and in unit sliding distance and is expressed as cubic millimeters per newton-meter.

4. RESULTS AND DISCUSSION

4.1. Friction Behavior

The typical coefficients of friction for the natural bulk diamond flat, the diamond films, and the DLC films in contact with a natural diamond pin and with Si_3N_4 pins that were obtained both in humid air (40 percent relative humidity) and in dry nitrogen are plotted as a function of the number of repeated passes in Figs. 1 and 2. The coefficients of friction given are typical, and the trends with number of passes are reproducible.

Comparative experiments were also conducted with the natural diamond flat, the diamond films, and the DLC films in contact with a hemispherical natural diamond pin and with Si_3N_4 pins in humid nitrogen at a relative humidity of approximately 40 percent in the same manner. The results

indicated that the friction characteristics of the natural diamond flat, the diamond films, and the DLC films in humid nitrogen were similar to those (Figs. 1 and 2) obtained in humid air. The differences in friction behavior between the humid air environment and the dry nitrogen environment may be primarily due to moisture differences in the environments. In this study therefore only humid air and dry nitrogen environments were considered.

Although different substrate materials were used in this investigation, the diamond films and the DLC films were thick enough and furthermore the adhesion strengths of the films to the substrates were great enough to screen the results from the effects of differences in substrate such as hardness. The friction and wear results presented in this paper were obtained from the diamond films deposited on silicon substrates and the DLC films deposited on Si_3N_4 substrates.

4.1.1. Natural Diamond Pin Contacts

With the natural diamond pin contacting the natural diamond flat (Fig. 1(a)), the coefficients of friction rapidly decreased with increasing number of passes and reached the equilibrium value of 0.01 or less. The moisture in the air seemed to have no effect on the coefficient of friction.

With the natural diamond pin contacting the diamond films (Fig. 1(b)), the initial coefficients of friction of the diamond films were 0.14 and 0.13, respectively, for the humid air and dry nitrogen environments. They rapidly decreased with increasing number of passes and reached the equilibrium values of 0.035 and 0.030, respectively. The moisture in the air did not affect the coefficient of friction. Comparing Fig. 1(b) with Fig. 1(a) shows that the friction behavior of the diamond film is similar to that of the natural diamond.

With the natural diamond pin contacting the DLC films (Figs. 1(c) and (d)), moisture markedly increased friction for both the low- and high-density DLC films deposited at 100-W and 240-W power, respectively. Furthermore, in humid air, the sliding action caused a breakthrough of both the low- and high-density DLC films and removed them from the contact areas at approximately 2500 and 5000 passes and above, respectively. In dry nitrogen, however, the DLC films did not wear off the substrates even after 10 000 passes. The coefficients of friction of the low- and high-density DLC films were generally low, and their friction behavior was similar to that of the natural diamond flat and the diamond films.

4.1.2. Si_3N_4 Pin Contacts

With the Si_3N_4 pins contacting the natural diamond flat (Fig. 2(a)), much higher coefficients of friction were measured in humid air than in dry nitrogen. In the presence of moisture the coefficient of friction remained constant with increasing number of passes. This relatively constant friction was the result of tribochemical interactions of the Si_3N_4 pin with the moisture. It is known that sliding action vastly increases the rate at which Si_3N_4 reacts chemically with moisture at the sliding interface.[18-20] The tribochemical interactions produce reaction products, such as silicon dioxide, at the sliding interface. These reaction products maintain the constant friction in this case.

With the Si_3N_4 pins contacting the diamond film (Fig. 2(b)), the initial coefficients of friction obtained were at 0.6 for both the humid air and dry nitrogen environments. The high initial coefficients of friction were probably due to the plowing and microcutting actions of the higher facet tips of the diamond grains. As sliding progressed, the coefficients of friction

decreased to approximately 0.1 and 0.02, respectively, at a total of 30 000 passes (Fig. 2(b)) for the humid air and dry nitrogen environments. The sharp decreases in friction at the start of the experiments suggest that wear occurred on the higher facet tips of the diamond grains in the earlier repeated passes and the tips became dull.

The final coefficients of friction μ_F for the Si_3N_4-pin-to-diamond-film contacts in humid air and in dry nitrogen were the same as those for the Si_3N_4-pin-to-natural-diamond-flat contacts (Figs. 2(a) and (b)). The fine-grain diamond films exhibited similar coefficients of friction as the natural diamond flat. Note that the coefficient of friction decreased and reached an equilibrium value of approximately 0.08 when sliding friction experiments were further extended to 35 000 to 40 000 repeated passes.

With the Si_3N_4 pins contacting the DLC films the coefficients of friction obtained in humid air for both low- and high-density DLC films were generally higher than those obtained in dry nitrogen as shown in Figs. 2(c) and (d). The moisture increased friction.

With the low-density DLC films (Fig. 2(c)) the coefficient of friction in humid air remained generally high and constant at about 0.17 to 2700 passes. And then a significant increase in the coefficient of friction began when the sliding action caused a film breakthrough at the contact area of the low-density DLC films at approximately 2700 repeated passes. After 2700 passes the coefficient of friction continued to increase and the DLC films wore off. In dry nitrogen, however, the coefficient of friction for the low-density DLC films also remained generally high and constant at about 0.15 over 10 000 passes, and the low-density DLC films did not wear off even after 10 000 passes, as shown in Fig. 2(c).

With the high-density DLC films (Fig. 2(d)) the coefficient of friction in humid air remained constant at about 0.2 over 10 000 passes. The high-density DLC films did not wear off even after 10 000 passes. In dry nitrogen the coefficient of friction for the high-density DLC films generally decreased with increasing number of passes. It became erratic and variable but low (0.02) after 2500 passes, as shown in Fig. 2(d).

4.2. Wear Behavior

Although the diamond films were somewhat abrasive in the initial stage of sliding friction experiments, the surfaces of the natural diamond flat, the diamond films, and the DLC films were generally not abrasive to the diamond pin and the Si_3N_4 pins either in humid air or in dry nitrogen.

The wear scars produced on the tips of the diamond pin and the Si_3N_4 pins in humid air and dry nitrogen were very smooth and had an extremely fine polished appearance. The wear tracks formed on the flat surfaces of the natural diamond, the diamond films, and the DLC films had a very smooth grooved appearance. Even, loose, fine wear debris existed mainly on the sides of wear scars of the natural diamond pin and the Si_3N_4 pins and on the sides of the wear tracks of the natural diamond flat, the diamond films, and the DLC films. The wear scars and wear tracks did not seem to be covered with wear debris. It is difficult to accurately measure the wear volume for the hemispherical tips of the diamond and Si_3N_4 pins. In this investigation therefore we attempted to estimate the average wear factors for the natural diamond flat, the diamond films, and the DLC films with the primary aim of generating specific wear factors that could be compared with those of other materials in the literature. The wear results will be discussed in the following section.

4.3. Comparison of Coefficients of Friction and Wear Factors

Figures 3(a) and (b) summarize the final coefficients of friction μ_F for the natural diamond flat, the diamond films, and the DLC films shown in Figs. 1 and 2.

Figures 4(a) and (b) present the average wear factors of the natural diamond flat, the diamond films, and the DLC films and 95-percent confidence limits for the average values. The data presented in Figs. 3 and 4 indicate the marked difference in friction and wear resulting from the combination of materials and environmental conditions.

4.3.1. Diamond Pin Contacts

Moisture in the environment seems to have no effect on either the coefficient of friction or the wear factor of the natural diamond flat and the diamond films in contact with the diamond pin (Figs. 3(a) and 4(a)). Both the coefficient of friction and wear factor of the diamond films were generally similar to those of the natural diamond flat both in humid air and in dry nitrogen. On the other hand, both the low- and high-density DLC films were susceptible to moisture: in particular, moisture increased both friction and wear (Figs. 3(a) and 4(a)). The wear factors of the materials in contact with the natural diamond pin were, in ascending order, natural diamond flat, diamond film, high-density DLC film, and low-density DLC film.

4.3.2. Si_3N_4 Pin Contacts

Moisture significantly changed the friction and wear of the natural diamond flat, the diamond films, and the DLC films in contact with the Si_3N_4 pins. Especially, the moisture increased the coefficients of friction for Si_3N_4 pins in contact with all the materials (natural diamond flat, diamond film, and DLC films). This suggests that the chemical nature of the Si_3N_4 pin

is important in the friction and wear of these materials. As mentioned earlier, the tribochemical interactions in humid air produce a reaction product, such as silicon oxides film, on the surface of the Si_3N_4 pin.[18-20] The increase in friction was due to the silicon oxides film produced on the surface of the Si_3N_4 pins in humid air.

With the natural diamond flat and the diamond films moisture increased friction but reduced wear (Figs. 3(b) and 4(b)). Shearing occurs in the silicon oxides film rather than in the natural diamond flat or in the diamond films during sliding. In humid air therefore the friction rose to a higher value (0.09 for the natural diamond flat and 1.1 for the diamond films). On the other hand, the silicon oxides film protected the surfaces of the natural diamond flat and the diamond films so that the wear factors for those materials were lower in humid air than in dry nitrogen.

With the low-density and high-density DLC films the moisture increased both friction and film wear (Figs. 3(b) and 4(b)). Especially, the wear factor of the low-density DLC films rose to extremely high values on the order of 10^{-5} $mm^3/N\text{-}m$. The high wear factor of the low-density DLC films was probably due to corrosive attack by the condensed moisture on the micropores and valleys of the surface irregularities.[10] It is interesting to note that in the dry nitrogen atmosphere the coefficients of friction of high-density DLC films were similar to those of the natural diamond flat and the diamond films. The wear factors of the materials in contact with the Si_3N_4 pins were, in ascending order, natural diamond flat, diamond film, high-density DLC film, and low-density DLC film. This order was the same as that for the natural diamond pin.

5. SUMMARY OF RESULTS

The following conclusions were drawn from the studies of the environmental effects on the friction and wear of a natural diamond flat, diamond films, and diamondlike carbon (DLC) films in contact with pin specimens of natural diamond and silicon nitride (Si_3N_4) in humid air and in dry nitrogen.

5.1 Natural Diamond Pin Contacts

1. Neither the coefficient of friction nor wear factor of the diamond films and the natural diamond flat were susceptible to moisture.

2. The coefficient of friction and wear factor of the diamond films were generally similar to those of the natural diamond flat both in humid air and in dry nitrogen.

3. The DLC films were much more susceptible to moisture. Moisture increased both the coefficient of friction and wear factor of the DLC films.

4. In dry nitrogen the coefficients of friction of both low- and high-density DLC films were generally low (0.02) and similar to those of the natural diamond flat and of the diamond films.

5. The wear factors of the materials investigated herein were, in ascending order, natural diamond flat, diamond film, high-density DLC film, and low-density DLC film.

5.2 Si_3N_4 Pin Contacts

1. Moisture in the environment increased the coefficients of friction for Si_3N_4 pins in contact with all the materials (natural diamond flat, diamond film, and DLC films). This increase in friction was due to the silicon oxides film produced on the surfaces of the Si_3N_4 pins in humid air.

2. In dry nitrogen the coefficients of friction of the high-density DLC films were generally variable but low (about 0.02) and similar to those of the natural diamond flat and the diamond films.

3. The coefficients of friction for low-density DLC films were generally high both in humid air and in dry nitrogen. Furthermore, the moisture greatly shortened the wear life of the low-density DLC films.

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FIGURE CAPTIONS

Figure 1. - Average coefficient of friction as function of number of passes of a natural diamond pin in contact with natural diamond flat, diamond film, and DLC films in humid air and in dry nitrogen.

(a) Natural diamond flat.

(b) Diamond film.

(c) Low-density DLC film deposited at 100 W.

(d) High-density DLC film deposited at 240 W.

Figure 2. - Average coefficient of friction as function of number of passes of silicon nitride pins in contact with natural diamond flat, diamond film, and DLC films in humid air and in dry nitrogen.

(a) Natural diamond flat.

(b) Diamond film.

(c) Low-density DLC film deposited at 100 W.

(d) High-density DLC film deposited at 240 W.

Figure 3. - Final coefficients of friction for natural diamond flat, diamond film, and DLC films in contact with a natural diamond pin and silicon nitride pins in humid air and in dry nitrogen.

Figure 4. - Wear factors for natural diamond flat, diamond film, and DLC films in contact with natural diamond pin and silicon nitride pins in humid air and in dry nitrogen.

TABLE 1.—CONDITIONS OF TRIBOEXPERIMENTS

Motion	Reciprocating
Load, N	1
Initial average Hertzian contact pressure, GPa	2.2 for natural diamond pin contacts and 0.91 for Si_3N_4 pin contacts
Sliding velocity, mm min^{-1}	86
Environment	Humid air; dry nitrogen
Temperature	Room temperature

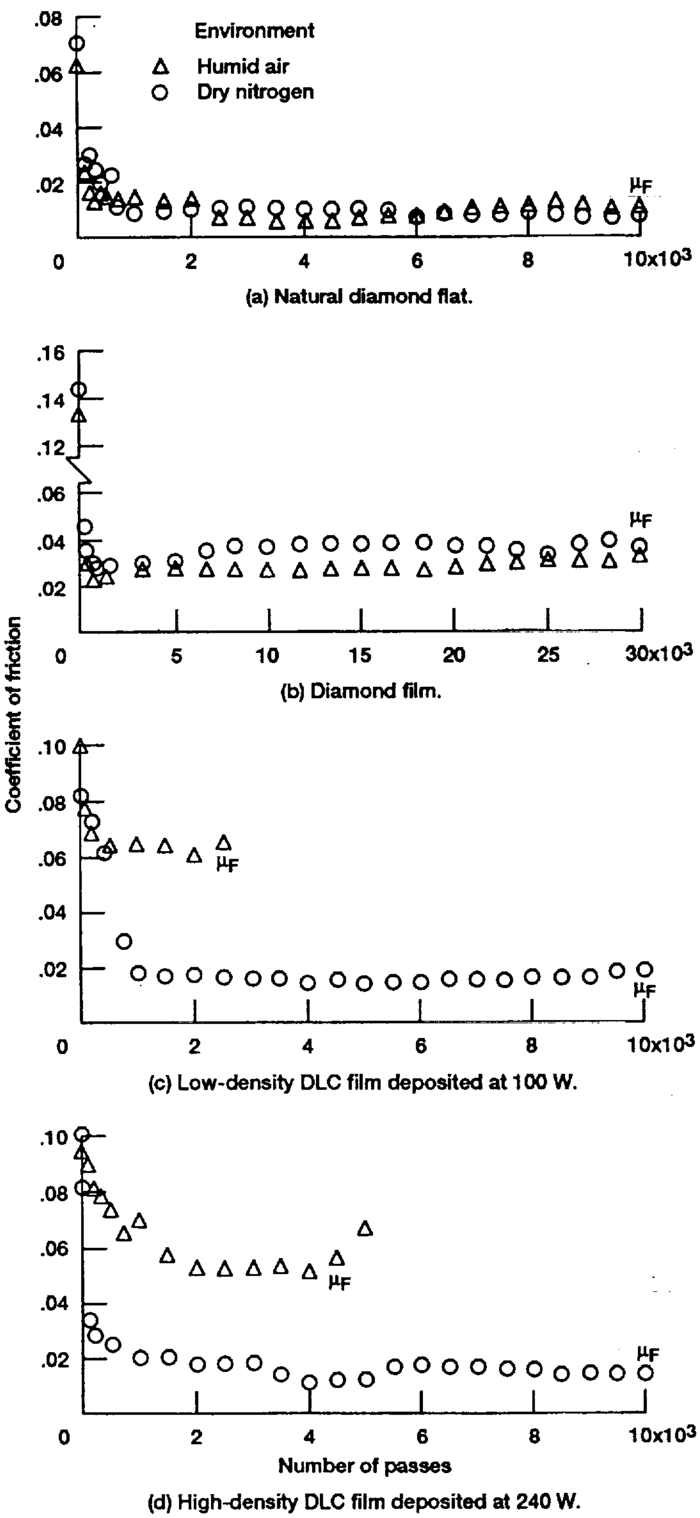


Figure 1.—Average coefficient of friction as function of number of passes of natural diamond pin in contact with natural diamond flat, diamond film, and DLC films in humid air and in dry nitrogen.

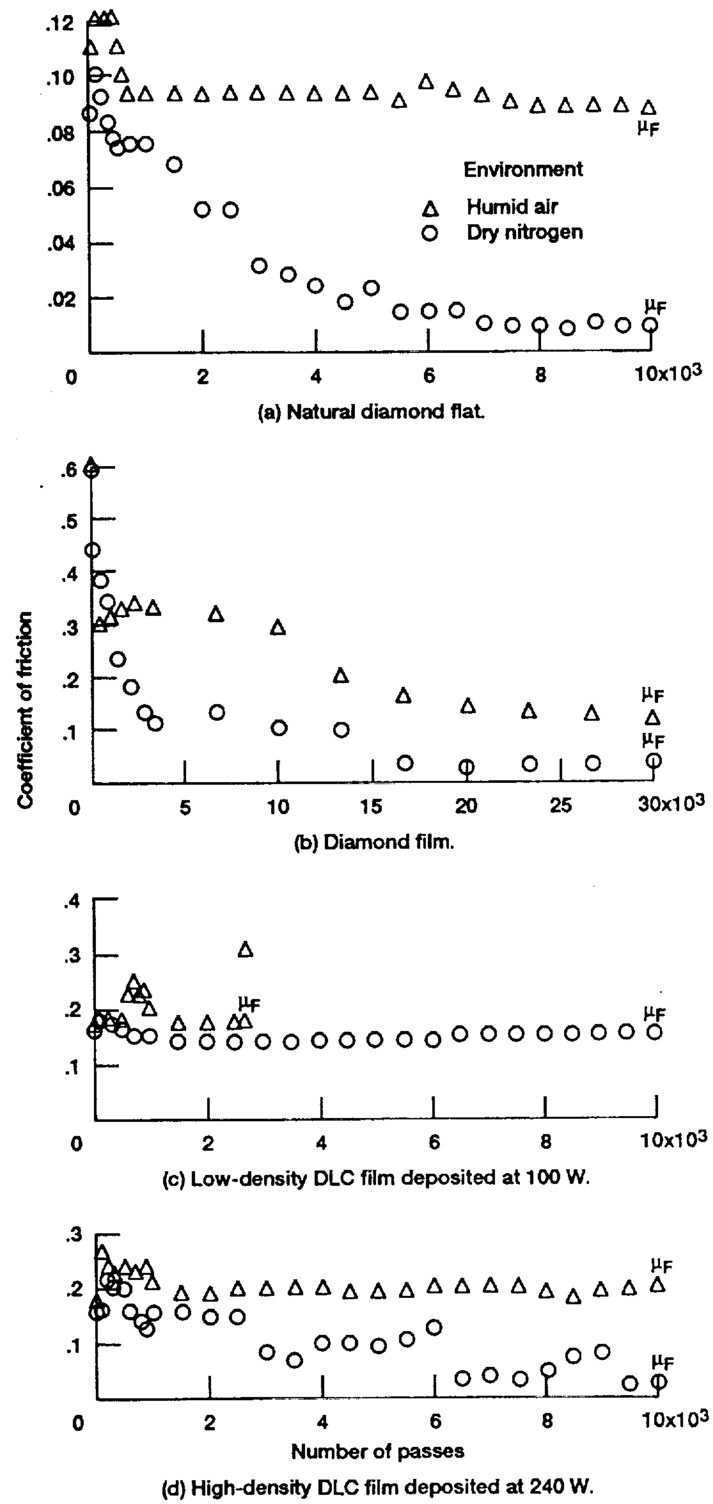
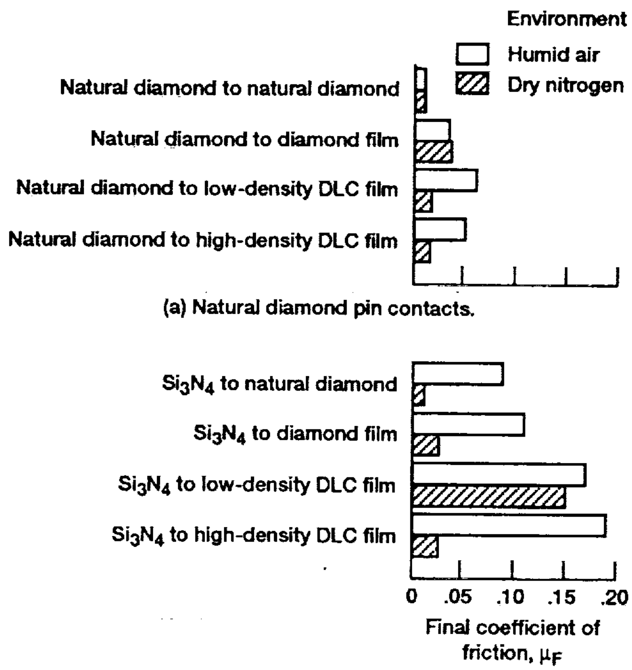
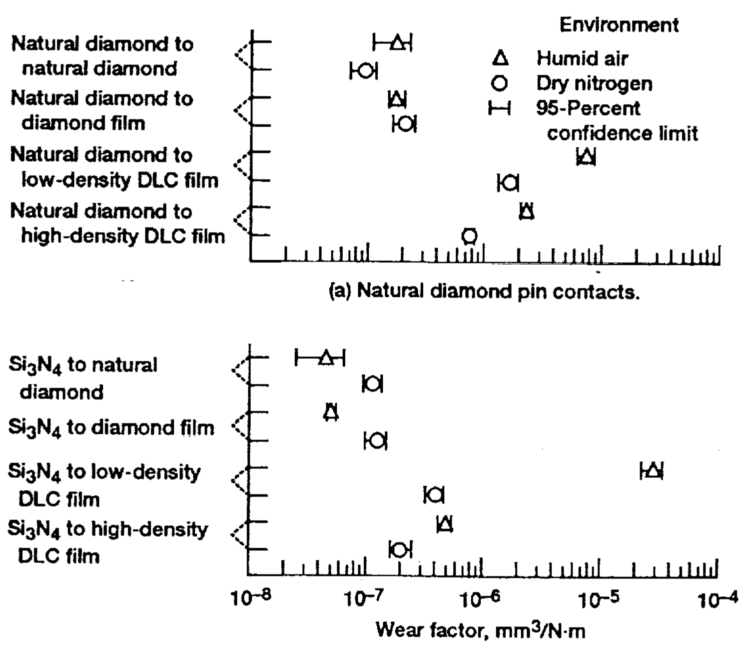


Figure 2.—Average coefficient of friction as function of number of passes of silicon nitride pin in contact with natural diamond flat, diamond film, and DLC films in humid air and in dry nitrogen.



(b) Silicon nitride pin contacts.

Figure 3.—Final coefficients of friction for natural diamond, diamond films, and DLC films in contact with a natural diamond pin and silicon nitride pins in humid air and in dry nitrogen.



(b) Silicon nitride pin contacts.

Figure 4.—Wear factors for natural diamond flat, diamond film, and DLC films in contact with a natural diamond pin and silicon nitride pins in humid air and in dry nitrogen.

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13. ABSTRACT ***(Maximum 200 words)***

Reciprocating sliding friction experiments were conducted with a natural diamond flat, diamond films, and low- and high-density diamondlike carbon (DLC) films in contact with pin specimens of natural diamond and silicon nitride (Si_3N_4) both in humid air and in dry nitrogen. The results indicated that for natural diamond pin contacts the diamond films and the natural diamond flat were not susceptible to moisture but that moisture could increase both the coefficients of friction and wear factors of the DLC films. The coefficients of friction and wear factors of the diamond films were generally similar to those of the natural diamond flat both in humid air and in dry nitrogen. In dry nitrogen the coefficients of friction of the high-density DLC films in contact with pin specimens of both diamond and Si_3N_4 were generally low (about 0.02) and similar to those of the natural diamond flat and the diamond films. The wear factors of the materials in contact with both natural diamond and Si_3N_4 were generally in the ascending order of natural diamond flat, diamond film, high-density DLC film, and low-density DLC film. The moisture in the environment increased the coefficients of friction for Si_3N_4 pins in contact with all the materials (natural diamond flat, diamond films, and DLC films). This increase in friction is due to the silicon oxide film produced on the surface of Si_3N_4 pins in humid air.

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